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A SELF-CONTAINED DOP PENETROMETER WITH A LIQUID-INJECTION AEROSOL GENERATOR.

by

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ABSTRACT

This report describes the design and construction details of an experimental model DOP penetrometer to be used in evaluating the efficiency of particulate filters and filter materials for protective masks. In the new design many of the deficiencies of the instrument presently used were eliminated.

The instrument includes a novel aerosol generator in which a constant flow at the desired concentration is attained by metering liquid dioctylphalate (DOP) from a hypodermic syringe with a infusion pump and spraying it into the generator by means of an electrostatic field.

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RESUME

Ce rapport décrit le concept et les détails de la construction d'un modèle experimental d'un pénétromètre DOP pour mesurer l'efficacité des filtres et des papiers à filtre contre la pénétration d'aérosols. On a corrigé beaucoup de défauts de l'instrument qu'on utilize présentement.

L'instrument comprend un nouveau générateur d'aérosols dans lequel on atteint un écoulement stable ayant la concentration voulue. On ajoute phtalate de di-octyl (DOP) en forme liquide d'une syringe hyopdermique entrainé par une pompe à infusion en utilizant un champ éléctrostatique.

INTRODUCTION

Background

High efficiency particulate filters and filter media were developed during World War II for use in protective masks and collective protectors. Shortly thereafter, the U.S. Atomic Energy Commission used filters of the same type in atomic energy installations to remove sub-micron radioactive particulate matter (1). Filters and filter media for both military and atomic energy applications are tested with an aerosol of the di(2-ethylhexy) ester of phthalic acid (dioctyl phthalate or DOP). For in situ testing or leak testing, the aerosol can be generated by atomizing liquid DOP. For evaluation purposes, a homogeneous aerosol of 0.3-µm-diameter particles is generated by condensing DOP vapor (2).

At DREO, a US Army Chemical Corps E27 Smoke Penetration Meter has been used to measure the removal of 0.3-µm DOP particles by filter paper, particulate filters for the protective mask canister and the complete canister. Later versions of this instrument were designated Penetrometer, Filter Testing, DOP, Q127 by the US Army.

These instruments have also been installed in industrial plants in Canada to monitor the production of protective mask filters and canisters and in paper mills to monitor the production of filter paper. Particularly at paper mills it was apparent that there was a need for a penetrometer that was readily portable, self-contained and operable after a relatively short warm-up period. Therefore, DREO has investigated the design and construction of an instrument that could be more easily used in industrial plants and also be suitable as a laboratory instrument.

Description of the Q 127

In the laboratory Q 127 penetrometer (3) smoke is produced by passing air over the surface of hot DOP. The Vapor-laden air is then mixed with a stream of cool air which causes the vapor to condense to form a smoke. Smoke concentration is determined by the temperature of the liquid DOP and the rate of air flow; particle size of the smoke is determined by the difference in temperature between the two air streams.

The liquid DOP is heated by means of an external heater regulated by a variable transformer and also by a controlled immersion heater which maintains the temperature at 172 ± 2°C. Warm-up time is decreased by an interval timer which bypasses the transformer and supplies full voltage to the jacket heater for a pre-determined time. The air flow (20 lpm) for vaporization of DOP is heated before entering the DOP container. The

diluent air flow (80 lmp) is pre-cooled by a heat exchanger where temperature is controlled by mixing hot and cold water. Particle size of the smoke is adjusted by varying the power to a heater located in the diluent stream just before the junction with the vapor stream. About 100 lpm of smoke is produced at a concentration of about 100 mg/m^3.

The smoke enters an aging chamber where it has time to stabilize. A vacuum pump draws a 5-lpm sample from the aging tank through the particle size analyzer and 32 lpm through the chuck and the light-scattering chamber of the penetration-measuring system. The remainder is vented to outside air.

The component under test is clamped in a chuck which is opened and closed by an air motor operated by push buttons. Movement of the bottom half of the chuck operates a foot valve causing the air which passes through the light-scattering chamber to be taken either from the chuck when it is closed or through a special filter when the chuck is open. While clean air is being passed through the scattering chamber, the vacuum control valve and flowmeter are bypassed in order to increase the flow rate and thus accelerate purging of the penetration measuring system of smoke.

A differential pressure gauge is connected to taps on the pipes entering and leaving the chuck. The resistance of the component under test is the differential pressure measurement less any blank reading obtained with an empty chuck.

Disadvantages of the Q 127

The Q 127 is not readily portable and is not self contained. Compressed air, vacuum and hot and cold water as well as electricity must be available where the penetrometer is to be used. It requires a long warm-up time to heat the liquid DOP and to stabilize the temperature.

The instrument is operated from a standing position and does not have a place to write data or stack items under test. The flow meters are located below the waist of the operator and are very difficult to read.

Since a blank reading for an empty chuck has to be measured and deducted from the resistance reading, a very large error could result when testing low-resistance filters.

Movement of the foot valve on closing the chuck causes a backlash of the differential pressure gauge which results in a considerable delay in making a resistance measurement.

The bypass of the vacuum control valve and flowmeter to increase flow during purging is neither necessary nor beneficial since the portion of the system between the test specimen and the foot valve, particularly the line to the differential pressure gauge, is not flushed. Initial

measurements made following a 100% calibration often show higher-than-average penetrations due to traces of smoke which have lingered in this section.

The principal objectives of the DREO development were to reduce the warm-up time, to increase the speed of response to particle size adjustments and to make the instrument self-contained except for electrical power. This report describes the design and construction of an experimental model of a smoke generator which meets these objectives. The main changes were to provide an air compressor and a vacuum pump and to redesign the smoke generator to produce DOP vapor by metering liquid DOP from a hypodermic syringe. A low-range differential pressure gauge was added and the chuck-operated foot valve was replaced with a micro-switch and solenoid valves to eliminate backlash of the differential pressure gauge. Improvements were also made for operational convenience to measurement of particle size, percent penetration and air-flow resistance but the basic principles used were not changed.

EXPERIMENTAL PENETROMETER

The Smoke Generator

DOP was metered into the smoke generator from a hypodermic syringe operated by a motor-driven injection pump. The liquid DOP passed through a hypodermic needle into the primary vaporizer where it was sprayed from the tip of the needle to a target by means of a 4000-V potential between them. The target consisted of a coil of resistance wire electrically heated to above the boiling point of DOP so that the DOP on striking it was immediately vaporized into a stream of air flowing through the vaporizer. The DOP vapor condensed and was carried along as a smoke of uncontrolled particle size. The smoke then entered a second vaporizer where it was again heated to vaporize the DOP. The vapor from the second vaporizer was mixed with a stream of cooler air which quenched the vapor to form a monodisperse smoke. (Fig. 1).

The particle size of the smoke was a function of the rate at which the vapor was cooled and varied with the temperature of the air after mixing the two streams. The vapor temperature was maintained by a proportional controller at a temperature just sufficient to revaporize all the primary smoke. To ensure that the quench air was cool enough to produce the 0.3-μm particles when the penetrometer was operated in very warm locations a thermo-electric cooler was installed ahead of the quench-air heater. Particle size was adjusted by manually regulating the power to the quench-air heater. The smoke was then further diluted with air at ambient temperature to the desired volume and concentration (Fig. 1).

In an earlier design, particle size was regulated by varying the temperature of the vapor stream while keeping the quench air temperature constant. The particle size appeared to be a function of the difference in temperature between the two streams. Increasing the vapor temperature produced smaller particles and decreasing the quench-air temperature also produced smaller particles. Later it was postulated that the large particles formed in the primary vaporizer were decreasing in size while passing through the second vaporizer but were not being completely evaporated. The size of these particles then varied with the vaporizer temperature and rate of flow through it. The DOP which had evaporated in passing through the vaporizer condensed to form smoke when cooled by the quench air and decreasing the quench temperature reduced the size of these particles. The smoke then contained two different particle sizes and particle size measurements were not valid.

To select a vaporizer temperature which would completely evaporate the primary smoke the vaporizer temperature was slowly increased while monitoring particle size until the particle size ceased to decrease and started to increase. This indicated that all of the primary smoke had been vaporized and that further heating was only raising the temperature of the air beyond the mixing point of the vapor and quench air streams which resulted in slower condensation of the vapor. A vapor temperature was then selected which was sufficiently high to vaporize all the primary smoke and particle size was regulated by varying the quench air temperature. Under these conditions the particle size varied with the temperature of the air in which condensation was taking place. The heater to regulate quench air temperature was located just before the point of mixing with the vapor. The heater was constructed of a nichrome wire element suspended along the axis of a glass tube. This provided a heater having a very low thermal capacity which gave fast response to power changes without overshooting.

The smoke generator was assembled on a standard aluminum chassis 17 in. x 14 in. x 3 in. fitted with a 19 in. x 12¼ in. front panel. This formed a drawer which slid into the right hand pedestal of the penetrometer cabinet. The DOP infusion pump, the high-voltage transformer and the thermoelectric cooler were mounted on top of the chassis. The high-voltage rectifiers and filter and the primary vaporizer were mounted below the chassis. Also below the chassis were a smoke-stabilizing chamber and an air-pressure regulator with the control knob extending up through the chassis. The secondary vaporizer and the quench-air heater were mounted above the chassis on a vertical plate fastened to the back of the chassis. The proportional temperature controller was fastened to the front panel and rested on the chassis. Also on the front panel were flow meters and valves to adjust the total air flow and the vapor air flow. The on-off switch, the high-voltage meter and variable transformer control were also on the front panel.

Primary Vaporizer

The primary vaporizer (Fig. 2) was housed in a brass ½-in. pipe tee. The air flow was straight through the tee using bushings to adapt it to the smoke-generator piping. The side arm was for the heater leads. The DOP entered through a hypodermic needle assembly which was screwed into the back of the tee opposite the heater.

The heater element was made by winding 18 cm of 19-Ω/ft resistance wire into a helix on a 1/8-in. mandrel and was embedded in procelain (Sauereisen cement) using a Teflon mold. The ends of the element were silver soldered to rigid wire leads which were pressed into holes through a piece of ½-in. Teflon rod threaded with ¼-in. pipe thread to fit the inside of a ½ x ¼ in. bushing in the side arm of the tee. The leads were connected to the 6.3-V winding on the transformer and one side was grounded.

The DOP was sprayed from a #27 hypodermic needle which was cemented into a ¼-in. bakelite rod threaded on the outside with a ¼-in. x 20 running thread so that the distance between the needle and heater could be adjusted. The high-potential side of a high-voltage power supply was connected to the needle. A non-conducting sleeve fitted over the tip improved the electrostatic spray performance. A baffle was installed in the inlet bushing to deflect the air stream so as not to blow the DOP spray away from the heater.

DOP Injection

The DOP was injected into the vaporizer by means of a Harvard infusion-withdrawal pump. The pump was operated with a 1-ml syringe (East Rutherford Syringe Co.) and a 1/5-rpm motor. The syringe contained 1 ml of DOP in a length of 2-1/8 inches. The pump lead screw had 24 threads per inch. This arrangement injected 0.004 g/min. of DOP to make 50 lpm of smoke at a concentration of 78 mg/m^3. At 0.3-µm diameter there would be about 5.8 x 10^6 particles per litre. A 1-ml filling of the syringe lasted about 4¼ h.

Power Supply for the Electrostatic Spray

The power used to spray the DOP and heat the target was supplied by a Hammond 216-60 transformer having a high-voltage winding of 3 kV at 110 V input and filament windings for 6.3 V, 0.6 A and 2.5 V, 1.75 A. The high voltage was half-wave rectified by twelve 1N 2071 diodes in series and filtered with a capacitance input filter to obtain a dc voltage equal to peak ac volts from the transformer. The transformer was operated from a 0-to-135-V variable transformer to obtain a dc potential of 0-5000 V which was applied between the hypodermic needle and vaporizing heater.

A resistance of twenty megohms in the high-voltage lead reduced the hazard of electrical shock. A fifty-microampere meter in series with a 100-megohm resistance was placed in the circuit to measure voltage.

At first, the 2.5-volt winding of the transformer was used for heating the vaporizer because of its lower voltage and higher current but it was connected internally to the high-voltage winding so that the heater had to be operated above ground and the hypodermic needle at ground potential. Since the pipe tee was also at ground potential the spacing between the needle and heater had to be very small to obtain the potential gradient around the needle tip to spray the DOP. At this small gap, arcing occured when the humidity was high and it was necessary to place a dryer in the air line to the vaporizer.

Later, the 6.3-V winding which was isolated from the high voltage was used to heat the vaporizer with one side of the heater being grounded. The high-voltage positive potential was applied to the hypodermic needle. A much better voltage gradient was obtained around the needle tip and it was possible to increase the distance between the needle and heater sufficiently to eliminate arcing even in high humidity allowing the dryer to be omitted.

A latch-in relay was installed to prevent surge in the high voltage when power was applied. The relay operated from a micro switch on the variable transformer so that the voltage setting had to be turned to zero volts to close the circuit (Fig. 3).

The Particle-Size Measuring System

Smoke for particle-size measurement was drawn from the smoke aging tank through the particle-size analyzer, flow meter and needle valve to the vacuum pump (Fig. 4). Except for details as noted, particle-size measurement was the same as in the Q 127 penetrometer. The particle-size analyzer consisted of a smoke chamber with a parallel beam of white light passing through it. The light scattered by the smoke at right angles to the beam was viewed by two photomultiplier tubes. Two Polaroid discs, one fixed and one rotatable, were positioned between the smoke chamber and each of the phototubes. The two discs closest to the smoke chamber were fixed with their axes of polarization at right angles to each other. The other pair of discs had their polarization axes parallel to each other and were geared so that they could be rotated together. The angle of rotation of their common polarization axis relative to that of one of the fixed discs was measured on a vernier protractor scale. At an angle setting of 29° the intensity of the light falling on the two photomultiplier (PM) tubes should be equal when the particle size is 0.3-μm (3). The outputs of the phototubes were applied to the particle-size meter to give a zero centre reading when the particle size was correct. A calibrating lamp inserted into the smoke chamber provided a source of unpolarized light

which was used with a polarization angle to 45° to match the gains of the phototubes before making measurements. To allow for any non-linearity in the gain of the PM tubes the intensity of the calibrating lamp should be the same as that of the light scattered by the smoke. With a polarization setting of 0° (one phototube in the dark with Polaroids at right angles and the other being illuminated with Polaroids parallel) the intensity of the calibrating lamp was adjusted to give the same reading of the particle-size meter as the smoke under the same conditions.

Changes in Electronics of Particle-Size Analyzer

When carrying out the calibration procedure for the particle-size analyzer the final step was to adjust the total gain so that rotating the polarizing vernier through one degree caused a meter deflection of 10 μA. Any change in the gain of the instrument necessitated a complete recalibration of the analyzer and the whole procedure had to be repeated until the error became negligible. A 10,000-Ω variable resistance was installed as a shunt across the meter so that the sensitivity could be adjusted after calibrating without changing the gain.

The intensity control for the calibrating lamp, a 50-ohm, 25-watt variable resistance in series with the lamp, did not always sufficiently reduce the illumination to match that of the smoke. It was rewired to act as a voltage divider so that the lamp voltage could be varied from zero to six volts.

The separate switches for the calibrating and operating lamps of the particle-size analyzer were replaced with a single-pole, double-throw switch with a centre-off position so that either could be put on but not both at the same time.

Electronic noise due to vibration of the particle-size indicator was reduced by mounting it and the air compressor and vacuum pump on rubber.

Filter Penetration and Resistance Measuring System

Smoke for penetration measurements passed from the aging tank through the filter holding chuck, the light scattering chamber and flow meter to the vacuum pump. A particulate filter after the light-scattering chamber removed smoke to prevent DOP despoits in the flow meter (Fig. 4).

Filter resistance measurements were made by means of a differential pressure gauge reading from 0 to 10 inches of water. For very low pressure drops, an inclined manometer reading from zero to one inch, graduated in hundredths of an inch, could be activated by a push button and solenoid valve.

As in the Q 127 instrument penetration was measured by comparing the intensity of light scattered from smoke passing the filter with that of unfiltered smoke. In the light-scattering chamber, a beam of light was focused to a point within the smoke and a photomultiplier tube viewed the light scattered forward in the direction of the incident beam. An opaque disc on the centre of the focusing lens prevented incident light from reaching the photomultiplier directly. When the chuck was closed on an empty filter holder, all of the smoke passed through and the gain of the photomultiplier was adjusted to give a meter reading of 100%. When the chuck was opened, clean filtered air flushed all smoke out of the scattering chamber and the meter was adjusted to zero with a stray light control. The chuck was then closed with a test filter in the holder and the meter measured the smoke passing the filter as percentage of the unfiltered smoke.

Opening and closing of the chuck was accomplished by means of an air motor actuated by push buttons. A microswitch operated by chuck movement actuated solenoid valves (Fig. 4), which caused smoke to flow through the chuck to the light-scattering chamber or supplied clean air for purging.

Chuck

In order that the chuck accommodate canisters, pleated filters and flat sheets of paper a holder was made for paper and filters which filled the same space between the chuck jaws as a canister. The holder was recessed on one side to hold a rubber-mounted, parallel-pleated filter, (Fig. 5) and when inverted presented a flat surface, having a standard-area opening for clamping flat paper in the chuck. Two interchangeable rubber-faced adapters were made to screw on the upper part of the chuck. One, for testing flat paper, had a standard-area opening to mate with the holder in the lower part of the chuck and the other with the opening large enough to bear on either the rim of a canister or the rubber periphery of the parallel-pleated filter.

Assembled Penetrometer

The experimental model of the penetrometer which is shown in Fig. 6 was housed in a double-pedestal console constructed of slotted angle and plywood. The smoke generator and air compressor were located in the pedestal on the right while the mechanical analyzer, percent-penetration scattering chamber and the vacuum pump were in the other. The air-operated chuck was located on the table surface above the kneehole and was mounted on the raised back of the cabinet. The particle size meter, the percent-penetration meter, flowmeters and differential pressure gauges were also mounted on the back of the cabinet. The unit was mounted on casters for mobility.

An electrical diagram for the penetrometer is given in Fig. 7.

PERFORMANCE

The experimental model penetrometer was independent of services other than electrical. It was more portable than the Q 127. It could be operated from a sitting position with meters and controls at eye level and provided a surface for writing and stacking test items. The smoke generator warm-up was short. When the quench-air cooler was not used, 0.3-μm smoke was produced in about three minutes from turning on the generator. On start-up, smoke from the primary vaporizer was produced almost immediately and reached the particle-size analyzer in about half a minute causing the indicator to swing far to the large-particle side. The secondary vaporizer then began to re-vaporize the primary smoke and the particle size decreased to 0.3-μm in about three minutes (Fig. 8). When the quench-air cooler was used an additional two to three minutes was required for it to come to equilibrium.

Response to adjustment to particle size was quite fast. When the sample was taken upstream of the aging chamber the particle-size indicator began to respond within fifteen seconds of making the adjustment. When the sample was taken from the aging chamber there was a lag of about thirty seconds.

The particle size was not as stable as in the Q 127 but could be maintained within the permissible limits of a meter deflection equivalent to rotating the analyzer ± 1/2° from the 29° setting (Fig. 8). In a prototype instrument the use of better-quality pressure- and flow-regulating components would be expected to reduce these variations in particle size.

Resistance measurements could be made more quickly than with the Q 127 since backlash of the differential pressure gauge was eliminated and measurements in the low range made with the inclined manometer were much more precise.

Although the smoke-generator warm-up time was greatly reduced the tube-type circuitry of the particle-size analyzer and penetration indicator still required a considerable time to stabilize before the instrument was operable.

Smoke particle-size regulation appears suitable for automatically maintaining the size by means of a feed back from the analyzer and circuitry to do this is being incorporated into a new analyzer design.

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3. H.W. Knudsen and Locke White. Naval Research Laboratory Report No. P-2642, September 1945.

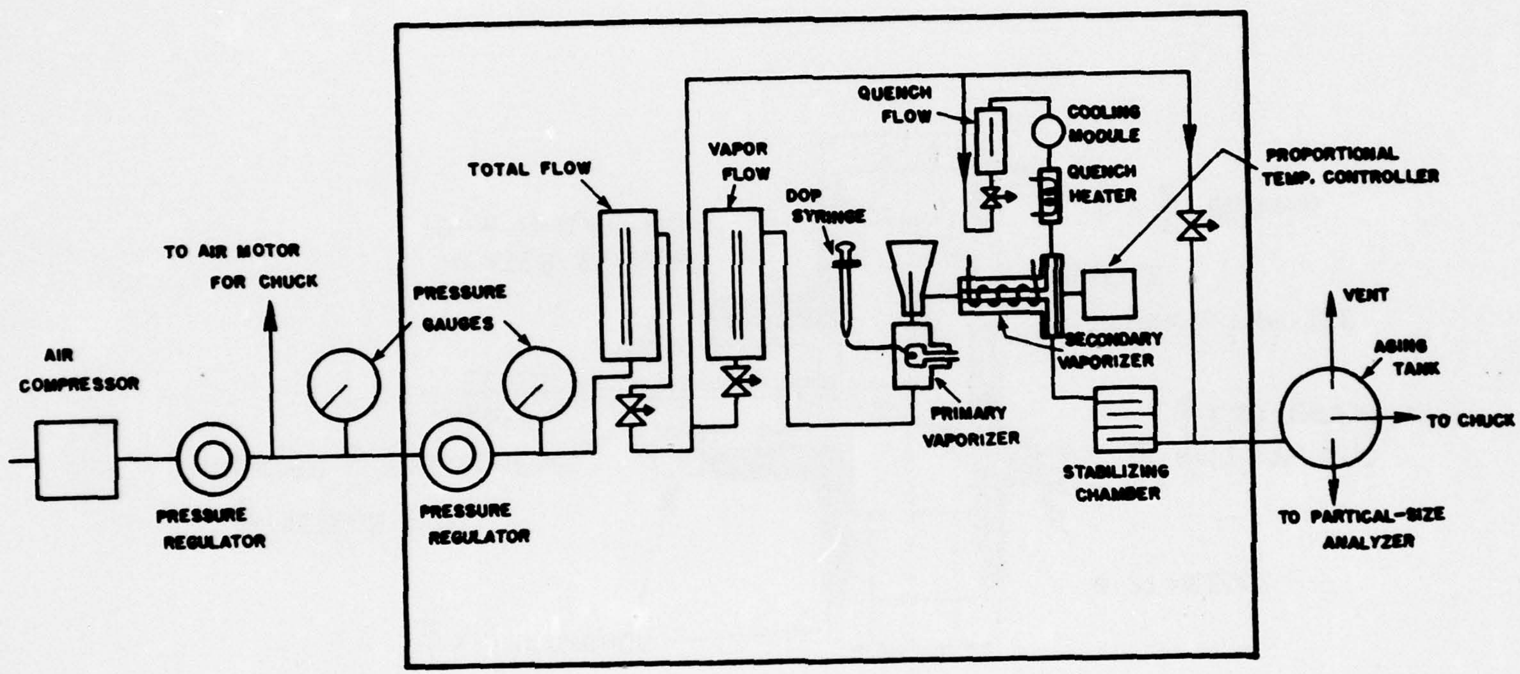

Figure 1. Flow diagram of smoke generator.

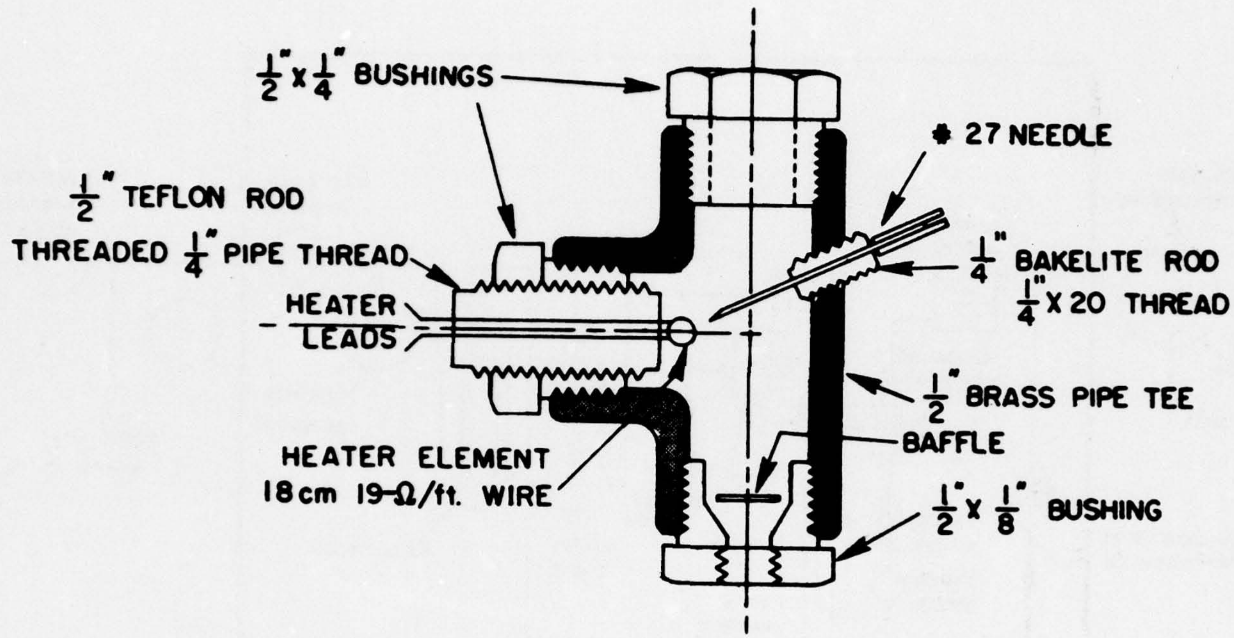

Figure 2. Detail of primary vaporizer.

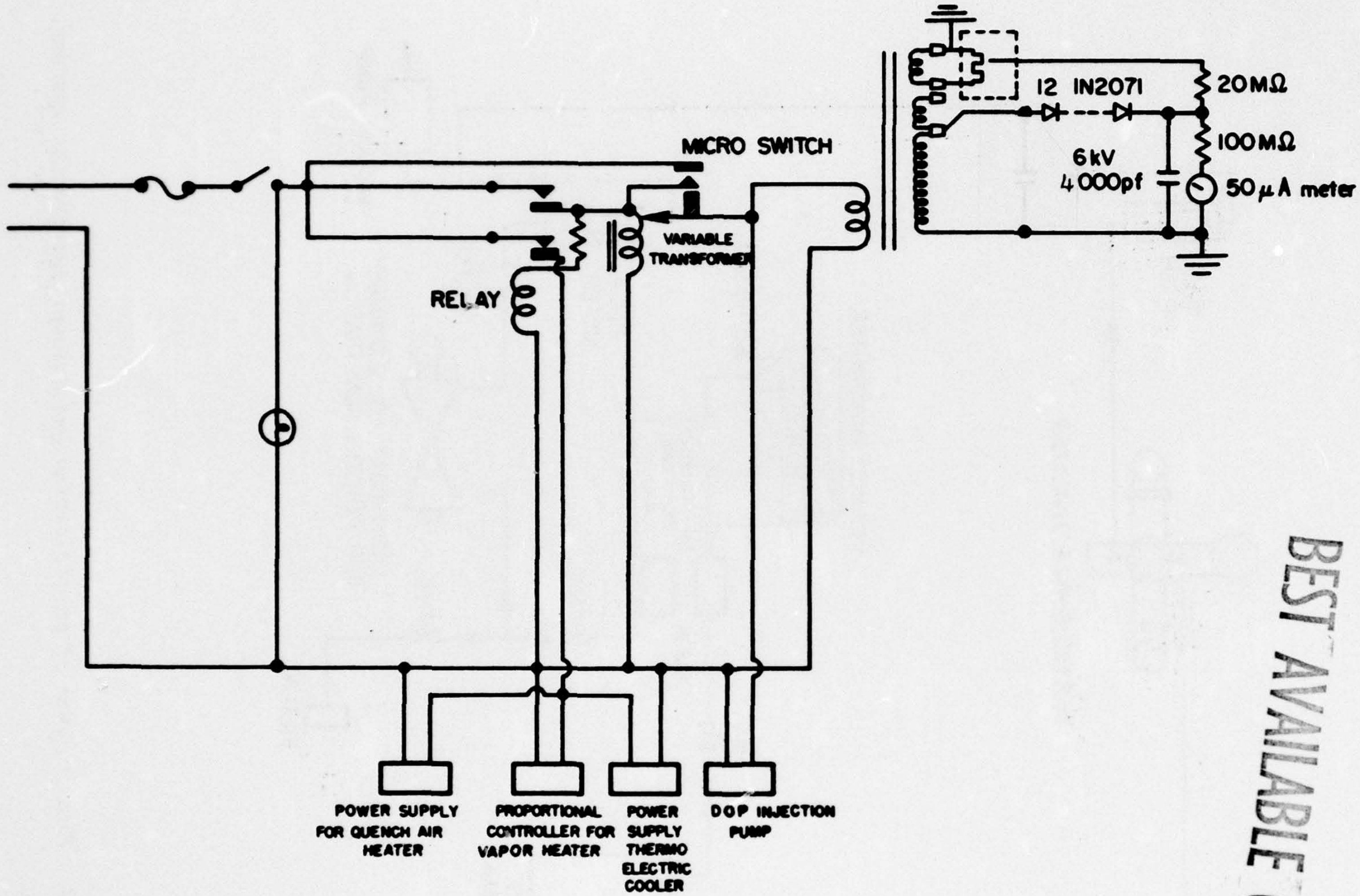

Figure 3. Smoke generator electrical circuits.

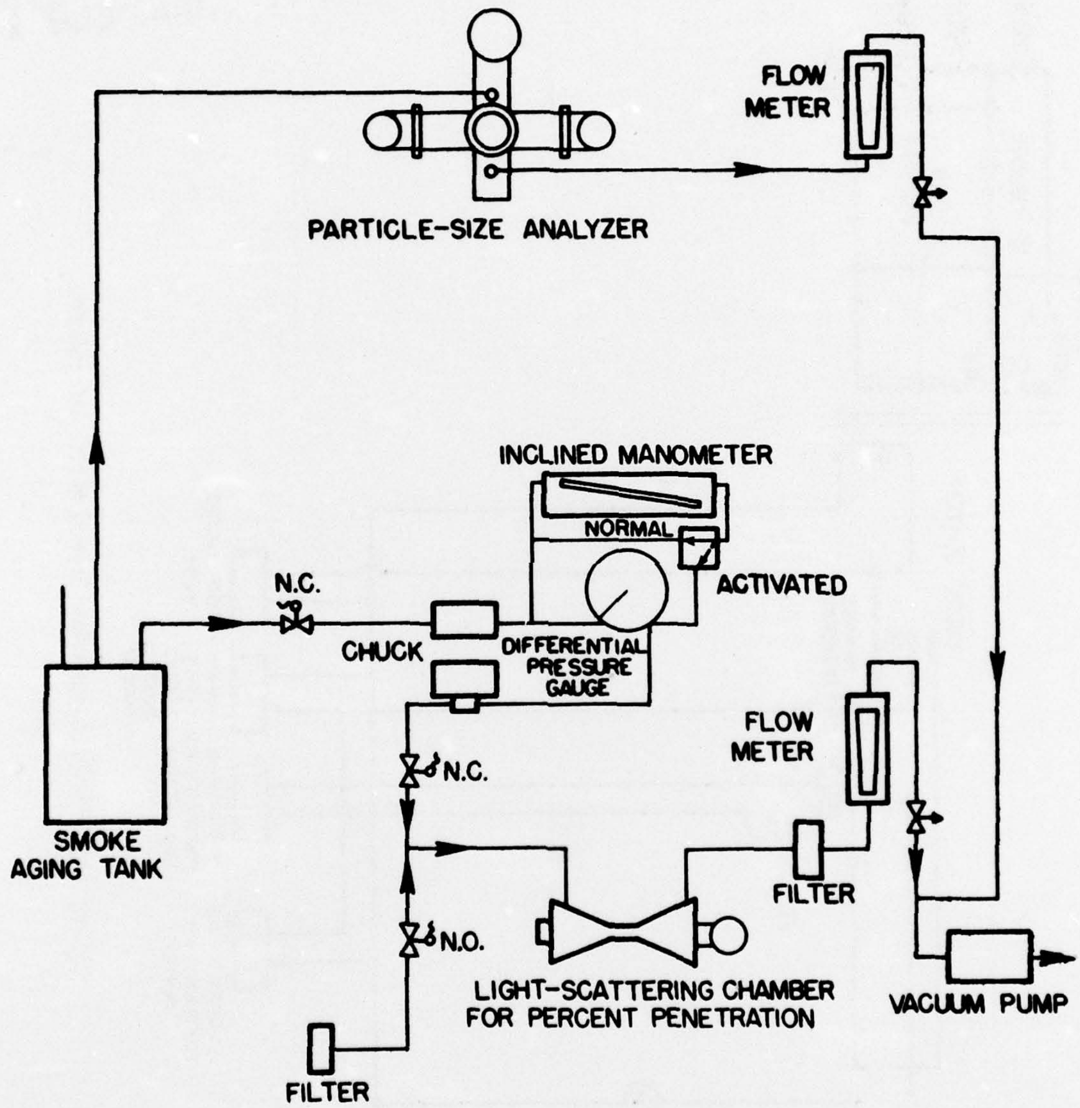

Figure 4. Flow diagram of particle-size and percent-penetration systems.

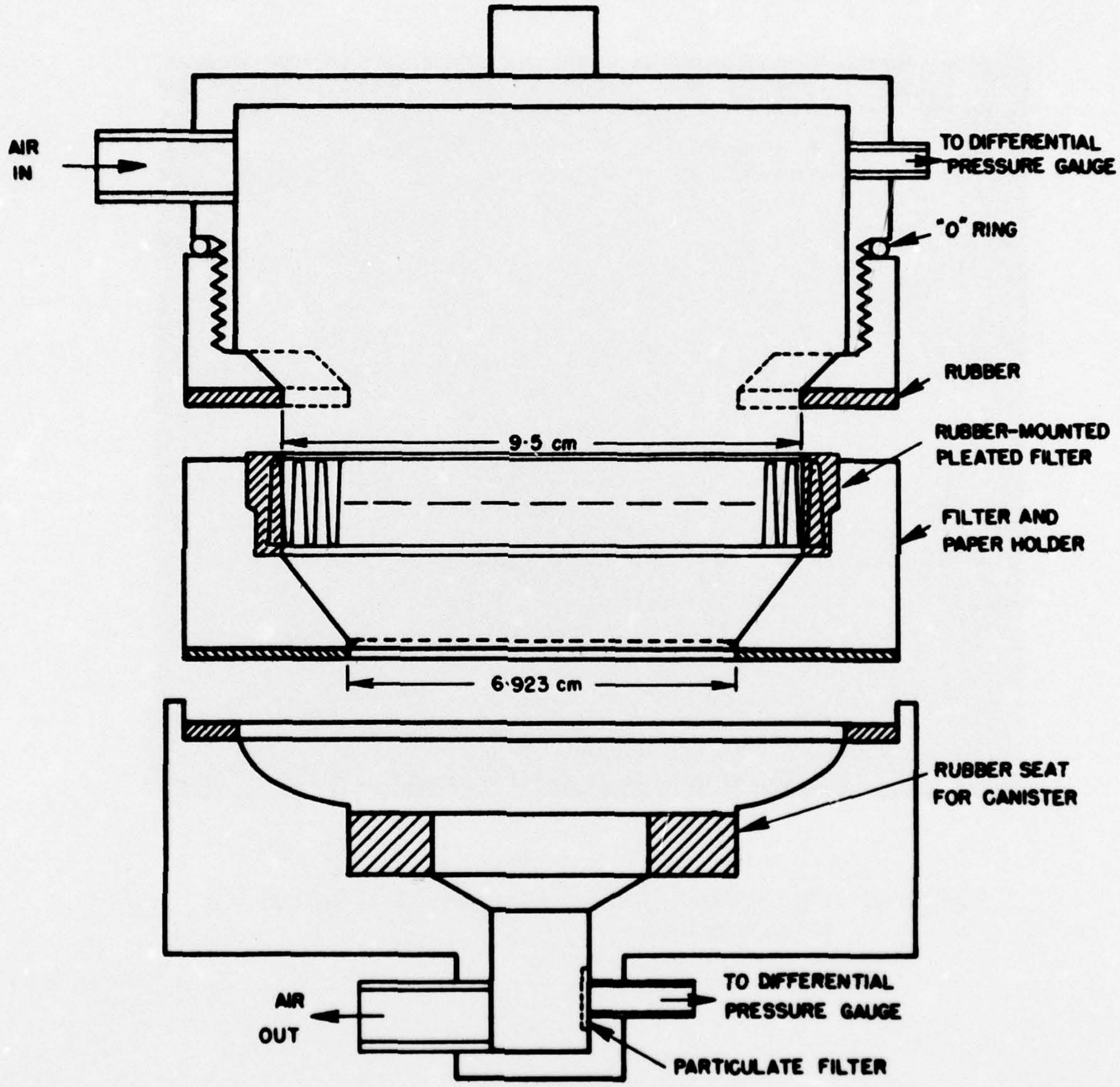

Figure 5. Chuck and adapter for pleated filters and papers.

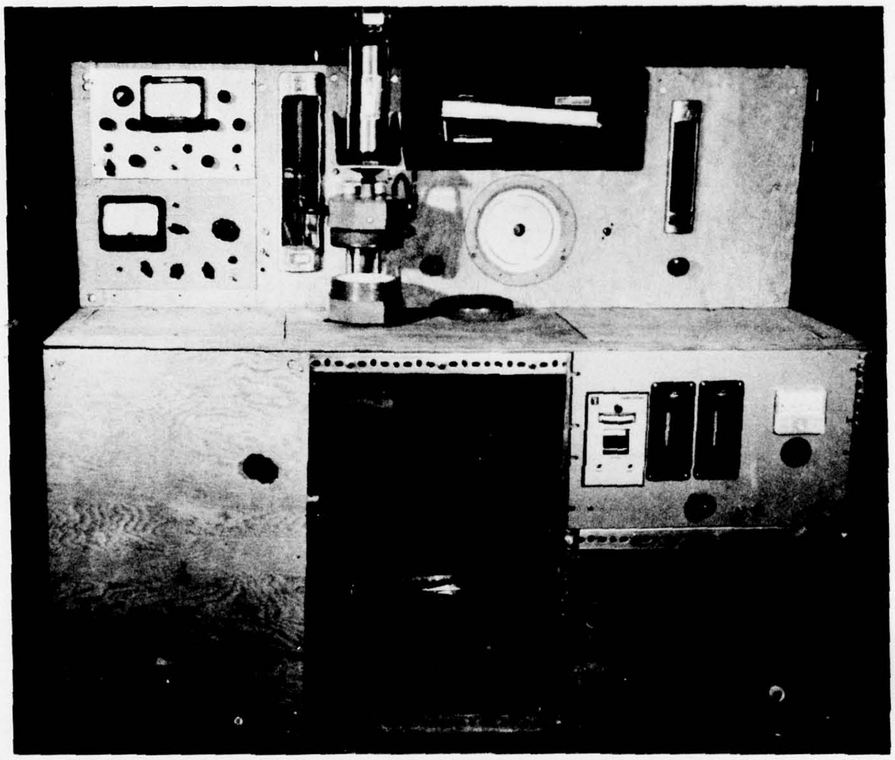

Figure 6. Photograph of the experimental model of the penetrometer.

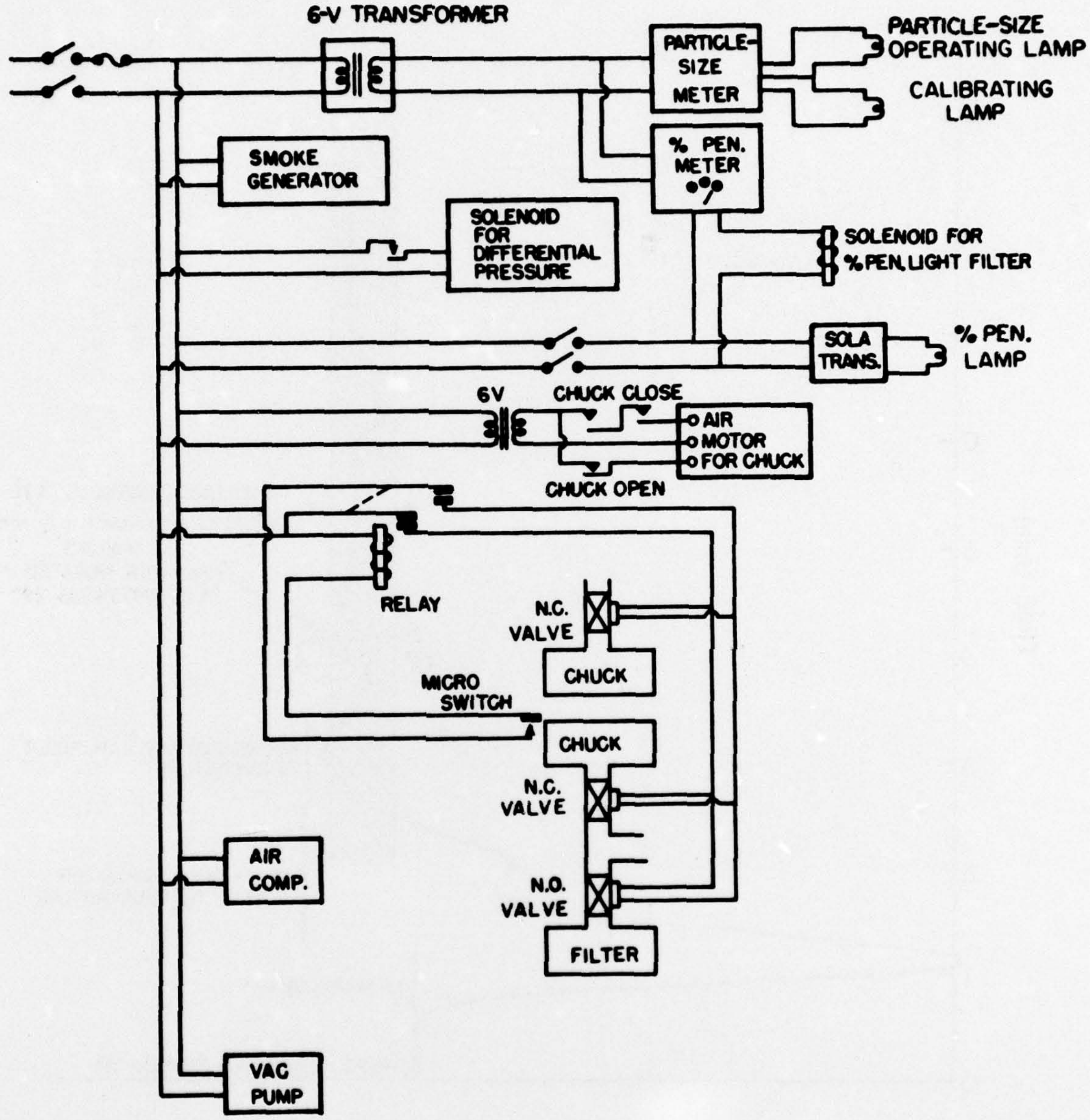

Figure 7. Penetrometer electrical circuits.

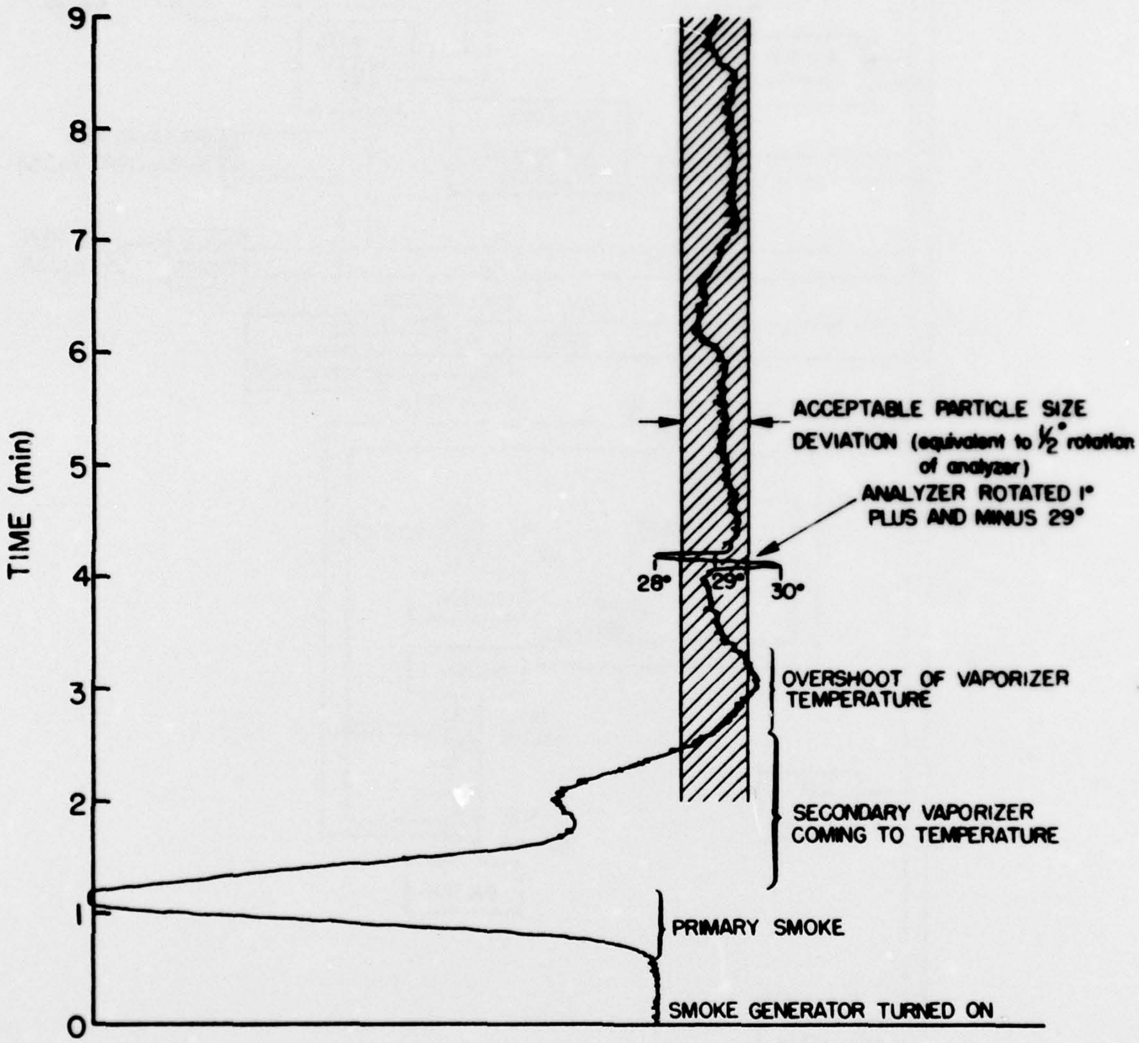

Figure 8. A recording of the output of the particle-size analyzer illustrating the start-up time and particle-size stability of the smoke generator.

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